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Synchrotron Infrared Photoacoustic Spectroscopy of Solids

K.H. Michaelian (Natural Resources Canada), R.S. Jackson (Bruker Optics Inc.), C.C. Homes (BNL)

Beamline(s): U10A

Introduction: Photoacoustic (PA) spectroscopy allows the characterization of condensed-phase materials with little or no sample preparation. In the infrared, this technique yields absorption spectra containing bands that arise from the functional groups present in a sample. PA infrared spectroscopy is thus suitable for a variety of analytical applications. Synchrotron (SR) infrared PA spectroscopy was implemented for the first time in this work.

Methods and Materials: An MTEC 100 PA cell was interfaced with the Bruker IFS 66 v/S infrared spectrometer at U10A. Mid- and far-infrared spectra were obtained for several carbonaceous samples (glassy carbon, carbon-filled rubber and hydrocarbon coke) and common clay. PA intensities obtained with SR were compared with those from a thermal source for a variety of beam sizes, selected by placing suitable brass apertures immediately above the sample in the PA cell [1].

Results: The ratio of the normalized SR spectrum to the globar spectrum was calculated as a function of wavenumber for each beam size. This ratio tends to be greater at longer wavelengths (lower wavenumbers), decreasing gradually at higher wavenumbers in each experiment. This phenomenon is illustrated in Fig. 1, which shows data obtained for glassy carbon with a 5-mm beam. The crossing frequency, defined as the wavenumber at which the SR/globar intensity ratio equals 1.0, is approximately 200 cm^{-1} under these conditions. The dependence of crossing frequency on aperture size is shown in Fig. 2; a linear fit of the data suggests that the entire mid-infrared region (up to 4000 cm^{-1}) is more intense in SR PA spectra for aperture (or sample) sizes up to 0.5 mm.

Conclusions: Photoacoustic infrared spectroscopy was shown to be feasible using SR instead of a thermal source. SR is the superior source in a spectral region that is a function of beam size. The high wavenumber limit of this region exhibits a power-law dependence on aperture size.

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References:

[1] R.S. Jackson, K.H. Michaelian, C.C. Homes, “Photoacoustic spectroscopy using a synchrotron light source”, in “Fourier Transform Spectroscopy”, OSA Technical Digest (Optical Society of America, Washington DC, 2001), pp. 161-163.

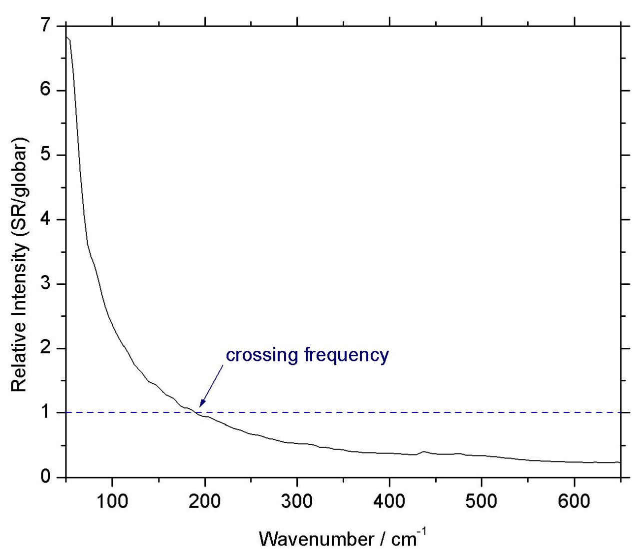

Figure 1. Ratio of the SR photoacoustic spectrum of glassy carbon to a similar spectrum obtained with a thermal source. The beam size was 5 mm.

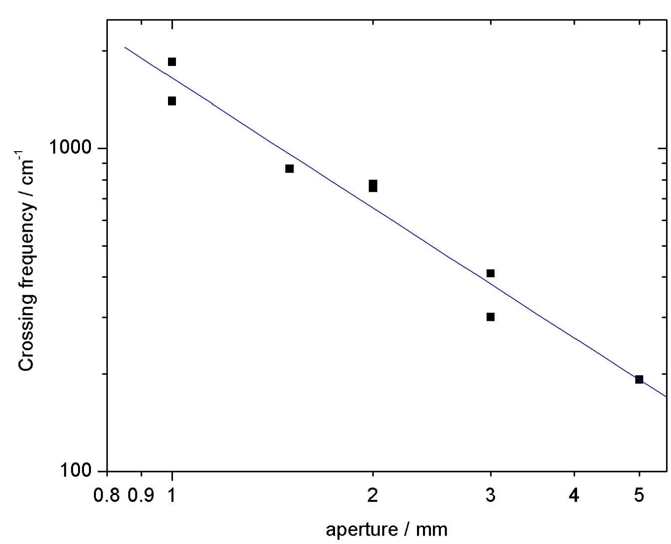

Figure 2. Dependence of crossing frequency on aperture size. The solid line is a least-squares fit to the data.